

Section B3. Sample Handling and Custody Requirements

Proper sample handling procedures for water, sediment, and biological samples are provided in Tables 6 and 7 (on the following pages), as well as in SOP's for Field Sample Collection provided in **Appendix D** (for all sample types except for biological assessment and benthic infaunal community assessment) and **Appendix G** (biological assessment and benthic infaunal community assessment QAPP's). Table 6 provides a summary of recommended sample containers, sample volumes, initial preservation, and maximum storage times for water samples. Table 7 provides the same information for bed sediment, tissue, and biota samples.

In the field, all samples will be packed in wet ice or frozen ice packs during shipment, so that they will be kept at approximately 4°C. Samples will be shipped in insulated containers. All caps and lids will be checked for tightness prior to shipping.

All samples will be handled, prepared, transported and stored in a manner so as to minimize bulk loss, analyte loss, contamination or biological degradation. Sample containers will be clearly labeled with an indelible marker. Where appropriate, samples may be frozen to prevent biological degradation. Water samples will be kept in Teflon™, glass, or polyethylene bottles and kept cool at a temperature of 4°C until analyzed. Maximum holding times for specific analyses are listed in Tables 6 and 7 on the following pages.

Ice chests are sealed with tape before shipping. Samples are placed in the ice chest with enough ice to completely fill the ice chest. RFA forms are placed in an envelope and taped to the top of the ice chest or they may be placed in a plastic bag and taped to the inside of the ice chest lid. It is assumed that samples in tape-sealed ice chests are secure whether being transported by staff vehicle, by common carrier, or by commercial package delivery. The receiving laboratory has a sample custodian who examines the samples for correct documentation, proper preservation and holding times.

Contract laboratories will follow sample custody procedures outlined in their QA plans. Contract laboratory QA plans are on file with the respective laboratory.

All samples remaining after successful completion of analyses will be disposed of properly. It is the responsibility of the personnel of each analytical laboratory to ensure that all applicable regulations are followed in the disposal of samples or related chemicals.

Chain-of-custody procedures require that possession of samples be traceable from the time the samples are collected until completion and submittal of analytical results. A complete chain-of-custody form is to accompany the transfer of samples to the analyzing laboratory.

Table 6. Summary of Sample Container, Volume, Initial Preservation, and Holding Time Recommendations for Water Samples

Parameters for Analysis in WATER Samples	Recommended Containers (all containers pre-cleaned)	Typical Sample Volume (ml)	Initial Field Preservation	Maximum Holding Time (analysis must start by end of max)
Conventional Constituents in Water				
Alkalinity	Polyethylene bottles (see NOTE ⁽¹⁾ below)	100 ml	Cool to 4°C, dark	14 days at 4°C, dark
Chloride (Cl), Sulfate (SO ₄) and Fluoride (F)	Polyethylene bottles (see NOTE ⁽¹⁾ below)	300 ml	Cool to 4°C, dark	28 days at 4°C, dark
Ortho-phosphate (OPO ₄)	Polyethylene bottles (see NOTE ⁽¹⁾ below)	150 ml	Cool to 4°C, dark	48 hours at 4°C, dark
Nitrate + Nitrite (NO ₃ + NO ₂)	Polyethylene bottles (see NOTE ⁽¹⁾ below)	150 ml	Cool to 4°C, dark	48 hours at 4°C, dark
Total Kjeldahl Nitrogen (TKN)	Polyethylene bottles (see NOTE ⁽¹⁾ below)	600 ml	Cool to 4°C, dark	Recommend: 7 days Maximum: 28 days Either one at 4°C, dark
Total Dissolved Solids (TDS)	Polyethylene bottles (see NOTE ⁽¹⁾ below)	1000 ml	Cool to 4°C, dark	7 days at 4°C, dark
Ammonia (NH ₃)	Polyethylene bottles (see NOTE ⁽¹⁾ below)	500 ml	Cool to 4°C, dark	48 hours at 4°C dark; if acidify, 28 days at 4°C, dark
Total Phosphorus (TPO ₄)	Polyethylene bottles (see NOTE ⁽¹⁾ below)	300 ml	Cool to 4°C, dark	28 days at 4°C, dark
(1)NOTE: The volume of water necessary to collect in order to analyze for the above constituents is typically combined in four 1-liter polyethylene bottles, which also allows enough volume for possible re-analysis and for conducting lab spike duplicates. This is possible since the same laboratory is conducting all of the above analyses; otherwise, individual volumes apply.				
Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC)	40 ml glass vial	40 ml (one vial)	Cool to 4°C, dark	28 days at 4°C, dark
Total Suspended Solids (TSS)	500 ml amber glass jar	1000 ml (two jars)	Cool to 4°C, dark	7 days at 4°C, dark
Suspended Sediment Concentration (SSC)	500 ml amber glass jar	500 ml (one jar)	Cool to 4°C, dark	7 days at 4°C, dark
Chlorophyll <i>a</i> Pheophytin <i>a</i>	1-L amber polyethylene bottle	1000 ml (one bottle)	Cool to 4°C, dark	Keep at 4°C, dark, but must filter within 48 hours. Filters may be stored frozen up to 30 days.

Parameters for Analysis in WATER Samples	Recommended Containers (all containers pre-cleaned)	Typical Sample Volume (ml)	Initial Field Preservation	Maximum Holding Time (analysis must start by end of max)
Non-Routine Compounds in Water Samples				
OIL AND GREASE	1-L glass jar with Teflon lid-liner, rinsed with hexane or methylene chloride	1000 ml (one jar)	Add 2 ml conc. H ₂ SO ₄ to pH <2; cool to 4°C, dark.	28 days at 4°C, dark
PHENOLS	1-L glass jar with Teflon lid-liner	1000 ml (one jar)	Add 2 ml conc. H ₂ SO ₄ to pH <2; cool to 4°C, dark.	28 days at 4°C, dark
CYANIDE	1-L cubitainer	1000 ml (one cubitainer)	Add 2 ml 1:1 NaOH to make pH > 12; Add 0.6 g ascorbic acid if residual Cl present. Cool to 4°C, dark.	14 days at 4°C, dark
BIOCHEMICAL OXYGEN DEMAND (BOD)	4-L cubitainer	4000 ml (one cubitainer)	Cool to 4°C, dark. Add 1g FAS crystals per liter, if residual Cl present.	48 hours at 4°C, dark
CHEMICAL OXYGEN DEMAND (COD)	1-L cubitainer	110 ml (one cubitainer)	Add 2 ml conc. H ₂ SO ₄ to make pH <2. Cool to 4°C, dark.	28 days at 4°C, dark
Trace Metals in Water Samples				
DISSOLVED METALS (except Dissolved Mercury)	60 ml polyethylene bottle, pre-cleaned in lab using HNO ₃	60 ml (one bottle) if salinity <0.5 ppt 180 ml (three bottles) if salinity >0.5 ppt	Filter at sample site using 0.45 micron in-line filter, or syringe filter. Cool to 4°C, dark. Acidify in lab, within 48 hrs, using pre-acidified container (ultra-pure HNO ₃) for pH<2.	Once sample is filtered and acidified, can store up to 6 months at room temperature
DISSOLVED MERCURY	250 ml glass or Teflon bottle, pre-cleaned in lab using HNO ₃	250 ml (one bottle)	Cool to 4°C, dark. Filter in lab within 48 hours, using bench top Hg filtration apparatus. Acidify in lab within 48 hrs, with pre-tested HCL to 0.5%.	Once sample is filtered and acidified, can store up to 6 months at room temperature

Parameters for Analysis in WATER Samples	Recommended Containers (all containers pre-cleaned)	Typical Sample Volume (ml)	Initial Field Preservation	Maximum Holding Time (analysis must start by end of max)
Trace Metals in Water Samples				
TOTAL METALS (except Total Mercury)	60 ml polyethylene bottle, pre-cleaned in lab using HNO ₃	60 ml (one bottle) if salinity <0.5 ppt 180 ml (three bottles) if salinity >0.5 ppt	Cool to 4°C, dark. Acidify in lab within 48 hrs, with pre-acidified container (ultra-pure HNO ₃), for pH<2.	Once sample is acidified, can store up to 6 months at room temperature
TOTAL MERCURY	250 ml glass or Teflon bottle, pre-cleaned in lab using HNO ₃	250 ml (one bottle)	Cool to 4°C, dark. Acidify in lab within 48 hrs, with pre-tested HCL to 0.5%.	Once sample is acidified, can store up to 6 months at room temperature.
HEXAVALENT CHROMIUM (filtered)	600 ml plastic or glass bottle	600 ml (one bottle)	Cool to 4°C, dark No acid	Keep at 4°C, dark for up to 24 hours; must notify lab in advance.
HARDNESS	200 ml polyethylene or glass bottle	200 ml (one bottle)	Cool to 4°C, dark OR Filter and add 2 ml conc. H ₂ SO ₄ or HNO ₃ to pH < 2; Cool to 4°C, dark.	48 hours at 4°C, dark 6 months at 4°C, dark
Synthetic Organic Compounds in Water Samples				
VOLATILE ORGANIC ANALYTES (VOA's) including VOC, MTBE and BTEX	40 ml VOA vials	120 ml (three VOA vials)	All vials are pre-acidified (50% HCl or H ₂ SO ₄) at lab before sampling. Cool to 4°C, dark	14 days at 4°C, dark
PESTICIDES & HERBICIDES* <input type="checkbox"/> Organophosphate Pesticides <input type="checkbox"/> Organochlorine Pesticides <input type="checkbox"/> Chlorinated Herbicides SEMI-VOLATILE ORGANICS* POLYCHLORINATED* BIPHENYL AND AROCHLOR COMPOUNDS TPH, PAH, PCP/TCP*	1-L I-Chem 200-series amber glass bottle, with Teflon lid-liner (per each sample type)	1000 ml (one container) *Each sample type requires 1000 ml in a separate container	Cool to 4°C, dark If chlorine is present, add 0.1g sodium thiosulfate	Keep at 4°C, dark, up to 7 days. Extraction must be performed within the 7 days; analysis must be conducted within 40 days.

Parameters for Analysis in WATER Samples	Recommended Containers (all containers pre-cleaned)	Typical Sample Volume (ml)	Initial Field Preservation	Maximum Holding Time (analysis must start by end of max)
Toxicity Testing Water Samples				
TOXICITY IN WATER	Four 2.25 L amber glass bottles	9000 ml	Cool to 4°C, dark	48 hrs at 4°C, dark
Bacteria and Pathogens in Water Samples				
<i>E. Coli</i>	Factory-sealed, pre-sterilized, disposable Whirlpak® bags or 125 ml sterile plastic (high density polyethylene or polypropylene) container	100 ml volume sufficient for both <i>E. coli</i> <u>and</u> <i>Enterococcus</i> analyses	Sodium thiosulfate is pre-added to the containers in the laboratory (chlorine elimination). Cool to 4°C; dark.	STAT: 6 hours at 4°C, dark if data for regulatory purposes; otherwise, 24 hrs at 4C, dark if non-regulatory purpose.
<i>Enterococcus</i>	Factory-sealed, pre-sterilized, disposable Whirlpak® bags or 125 ml sterile plastic (high density polyethylene or polypropylene) container	100 ml volume sufficient for both <i>E. coli</i> <u>and</u> <i>Enterococcus</i> analyses	Sodium thiosulfate is pre-added to the containers in the laboratory (chlorine elimination). Cool to 4°C; dark.	STAT: 6 hours at 4°C, dark if data for regulatory purposes; otherwise, 24 hrs at 4C, dark if non-regulatory purpose.
FECAL COLIFORM	Factory-sealed, pre-sterilized, disposable Whirlpak® bags or 125 ml sterile plastic (high density polyethylene or polypropylene) container	100 ml volume sufficient for both fecal <u>and</u> total coliform analyses	Sodium thiosulfate is pre-added to the containers in the laboratory (chlorine elimination). Cool to 4°C; dark.	STAT: 6 hours at 4°C, dark if data for regulatory purposes; otherwise, 24 hrs at 4C, dark if non-regulatory purpose.
TOTAL COLIFORM	Factory-sealed, pre-sterilized, disposable Whirlpak® bags or 125 ml sterile plastic (high density polyethylene or polypropylene) container	100 ml volume sufficient for both fecal <u>and</u> total coliform analyses	Sodium thiosulfate is pre-added to the containers in the laboratory (chlorine elimination). Cool to 4°C; dark.	STAT: 6 hours at 4°C, dark if data for regulatory purposes; otherwise, 24 hrs at 4C, dark if non-regulatory purpose.

Table 7. Summary of Sample Container, Volume, Preservation, and Storage Requirements for SWAMP Bed Sediment, Biota, and Tissue Samples (for contaminant analysis)

Parameters for Analysis	Recommended Containers	Typical Sample Volume (ml)	Initial Field Preservation	Maximum Holding Time
Bed Sediment Samples				
Trace Metals, including Hg and As (except for Se--see below)	60 ml I-Chem 300-series clear glass jar with Teflon lid-liner; Pre-cleaned	60 ml (one jar)	Cool to 4°C, dark, up to 14 days	12 months ⁽¹⁾ (-20°C)
Selenium (separate container required)	60 ml I-Chem 300-series clear glass jar with Teflon lid-liner; Pre-cleaned	60 ml (one jar)	Cool to 4°C, dark, up to 14 days	12 months ⁽¹⁾ (-20°C)
Synthetic Organic Compounds	250 ml I-Chem 300-series amber glass jar with Teflon lid-liner; Pre-cleaned	500 ml (two jars)	Cool to 4°C, dark, up to 14 days	12 months ⁽¹⁾ (-20°C)
Sediment TOC	125 ml ⁽³⁾ clear glass jar; Pre-cleaned	125 ml (one jar)	Cool to 4°C, dark, up to 28 days	12 months ⁽²⁾ (-20°C)
Sediment Grain Size	125 ml ⁽³⁾ clear glass jar; Pre-cleaned	125 ml (one jar)	Cool to 4°C, dark, up to 28 days	28 days (4°C) <u>Do not freeze</u>
Sediment Toxicity Testing	1-Liter I-Chem wide-mouth polyethylene jar with Teflon lid-liner; Pre-cleaned	2-Liters (two jars filled completely)	Cool to 4°C, dark, up to 14 days	14 days (4°C) <u>Do not freeze</u>
<p>(1) Sediment samples for parameters noted with one asterisk (*) may be refrigerated at 4°C for up to 14-days maximum, but analysis <u>must</u> start within the 14-day period, or the sediment sample <u>must</u> be stored frozen at minus (-) 20°C for up to 12 months maximum.</p> <p>(2) Sediment samples for sediment TOC analysis can be held at 4°C for up to 28 days, and <u>should</u> be analyzed within this 28 day period, but can be frozen at any time during the initial 28 days, for up to 12 months maximum at minus (-) 20°C.</p> <p>(3) Sediment samples for TOC AND grain size analysis can be combined in one 250 ml clear glass jar, and sub-sampled at the laboratory in order to utilize holding time differences for the two analyses. If this is done, the 250 ml combined sediment sample must be refrigerated only (<u>not frozen</u>) at 4°C for up to 28 days, during which time the sub-samples must be aliquoted in order to comply with separate storage requirements (as shown above).</p>				

Table 7 (continued).

Summary of Sample Container, Volume, Preservation, and Storage Requirements for SWAMP Bed Sediment, Tissue (for contaminant analysis), and Biota Samples

Parameters for Analysis	Recommended Containers	Typical Sample Volume (ml)	Initial Field Preservation	Maximum Holding Time
Tissue samples				
Fish, crab, and shellfish tissue (for contaminant analysis)	Polyethylene bags (Teflon™ sheets in Ziplock™ bags); or glass (with Teflon™ lid); or polyethylene jar for trace metals sample only	200g	Freeze until processing	12 months (-20°C)
Biota (Benthic macroinvertebrates)				
FRESHWATER	plastic or glass	variable	90% ethyl alcohol OR 70% isopropyl alcohol OR Add formalin to produce a 5-10% formalin solution Store in dark and away from extremes of hot and cold.	5 years
MARINE	plastic or glass	variable	Add formalin buffered with borax to create a 10% formalin solution. After 2 weeks, sort sample and preserve with 70% ethanol	5 years
Netplankton	amber plastic or glass (Lugol's solution will permeate plastic cubitainers and stain materials in contact with cubitainer)	variable	Rinse net bucket with 3-5% buffered formalin OR If net bucket rinsed with tap water, preserve sample with 1 ml of modified Lugol's solution per 100 ml of sample. Store in dark and away from extremes of hot and cold.	5 years

Parameters for Analysis	Recommended Containers	Typical Sample Volume (ml)	Initial Field Preservation	Maximum Holding Time
Nannoplankton	amber plastic or glass (Lugol's solution will permeate plastic cubitainers and stain materials in contact with cubitainer)	500 g	1 ml of modified Lugol's solution per 100 ml of sample. Store in dark and away from extremes of hot and cold.	5 years
Nekton	plastic or glass	variable	Fix in a 10% formalin solution. After about 1 week thoroughly wash and preserve in 40 % ethyl alcohol Store in dark and away from extremes of hot and cold.	5 years

A sample is considered under custody if:

- it is in actual possession;
- it is in view after in physical possession;
- it is placed in a secure area (accessible by or under the scrutiny of authorized personnel only after in possession)

Field Log

Field crews shall be required to keep a field log for each sampling event. The following items should be recorded in the field log for each sampling event:

- date and time of sample collection, name of sampler(s), waterbody name
- sample ID numbers, including etched bottle ID numbers for Teflon™ mercury sample containers and unique IDs for any replicate or blank samples;
- the results of any field measurements (temperature, D.O., pH, conductivity, turbidity) and the time that measurements were made;
- qualitative descriptions of relevant water conditions (e.g. color, flow level, clarity) or weather (e.g. wind, rain) at the time of sample collection;
- a description of any unusual occurrences associated with the sampling event, particularly those that may affect sample or data quality.

The field crews shall have custody of samples during field sampling. Chain of custody forms will accompany all samples during shipment to contract laboratories. All water quality samples will be transported to the analytical laboratory directly by the field crew or by overnight courier.

Laboratory Custody Log

Laboratories shall maintain custody logs sufficient to track each sample submitted and to analyze or preserve each sample within specified holding times.